



Comparison of dead-end ultrafiltration behaviour and filtrate quality of sugar beet juices obtained by conventional and “cold” PEF-assisted diffusion

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ARTICLE INFO

Article history:

Received 4 March 2011

Received in revised form 2 May 2011

Accepted 6 May 2011

Available online 13 May 2011

Keywords:

Sugar beet juice

Extraction

Pulsed electric field

Ultrafiltration

Purification

Fouling

ABSTRACT

This work is devoted to ultrafiltration of sugar beet juices obtained by conventional high temperature diffusion and novel pulsed electric field (PEF) assisted low temperature diffusion. Filtration experiments were carried out in the concentration mode (up to the final volume concentration factor 7.5) using a batch dead-end filtration cell and hydrophilic polyethersulfone membranes with nominal molecular weight cut-offs (MWCO) of 10, 30 and 100 kDa. Two types of filtration experiments were carried out: without stirring and under constant stirring of feed juice. The transmembrane pressure was within the range 0.5–3.7 bar. The influence of filtration conditions (transmembrane pressure, membrane MWCO and stirring) on filtration rate, filtration mechanism and filtrate quality (purity and coloration) were studied and the results for juices obtained by conventional diffusion and by PEF-assisted diffusion were compared.

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1. Introduction

Membrane filtration of various bio-suspensions and extracts was studied intensively during the last decade [1–8]. It became commonly accepted that membrane filtration is a suitable method for clarification of natural juices from colloidal impurities (intracellular pectins, proteins, oligosaccharides and cell debris) since it decreases juice turbidity without any negative effect on juice's nutritional quality [9–13]. Ultrafiltration was tested as a tool for purification of raw and partially clarified sugar beet and sugar cane extracts [14–22]. It was shown that ultrafiltration may decrease concentration of non-sucrose compounds and colorants in the extract and increase extract purity [3,23–27].

However, filtration of natural juices is limited by membrane fouling, which results in decrease of filtration rate. Membrane fouling is attributed to pore blocking and formation of a polarized gel layer from colloidal impurities and polymer molecules contained by raw juice [1,2,7,8,12,28–31]. Formation of the gel layer substantially increases the total hydraulic resistance and decreases efficiency of filtration. However, it was reported that gel layer also may serve as a dynamic membrane, which enhances filtrate purity [18,29]. It was observed that the role of gel layer in separation of

impurities may be more important than the role of pore size of the membrane used for ultrafiltration [32].

The influence of preliminary juice clarification (removal of pectins, proteins, cell debris) on ultrafiltration was studied [33–40]. Several methods (including centrifugation, enzyme treatment, flocculants, adsorbents and filter aids) were used for juice clarification before ultrafiltration [33–40]. It was observed that application of these methods results in removal of polymers and cell debris and enhances filtration rate.

Preparation of sugar beet juice (diffusion juice) by conventional industrial method implies water extraction (diffusion) of sugar beet cossettes at a high temperature $T = 70\text{--}75\text{ }^{\circ}\text{C}$ [41,42]. High temperature leads to breakage of cell membranes and permits effective diffusion of sucrose through the beet tissue into the extracting water [41,42]. However, it also results in a thermal degradation of the cell walls and undesirable extraction of high molecular weight cell compounds (generally, pectin and protein). Pectin and other impurities decrease the quality of diffusion juice and complicate tremendously the process of sugar beet juice purification [30,33,43]. High concentration of pectins decreases efficiency of sugar beet juice ultrafiltration since pectin forms a polarized gel layer on the membrane surface [44].

During the last decade, great attention was paid to development of non-thermal methods of vegetable tissue processing based on the pulsed electric field (PEF) treatment [45]. Application of PEF with field intensity of 100–1000 V/cm and impulse duration from several microseconds to several milliseconds results in

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effective damage of the cell membranes and increases permeability of cells for small molecules or even some macromolecules [45]. However, PEF is not destructive to the cell walls of plant materials. It enables effective and more selective diffusion of low molecular weight intracellular components even at low temperatures [45].

It was shown recently that pre-treatment of sugar beet cossettes by pulsed electric field allows decrease of diffusion temperature from 70 °C to 30 °C [46]. The purity of the juice obtained at low temperature (30 °C) was not lower than the purity of the juice obtained by conventional thermal diffusion (at 70 °C) [46–48]. Moreover, the low temperature of diffusion did not result in degradation of cell walls and solubilization of pectins or other polymers. Therefore, concentration of colloidal substances in the “cold” sugar beet juice obtained at 30 °C was much lower than in juice obtained at 70 °C [47]. PEF-assisted “cold” diffusion at 30 °C also resulted in lower concentration of various colorants and their intermediates in the diffusion juice [47]. These factors may be advantageous for purification of the diffusion juice by means of ultrafiltration.

Our present work is devoted to ultrafiltration of sugar beet juice obtained by PEF-assisted “cold” diffusion at 30 °C. The aim of this work is to compare the mechanism of filtration and filtrate quality of sugar beet juices extracted at 30 °C and 70 °C, and to study the influence of filtration mechanism on the filtrate quality.

2. Materials and methods

2.1. Diffusion of sugar beet juices

The juices were obtained from fresh sugar beet roots (*Beta vulgaris*) supplied by Tereos (Chevrières, France). All the diffusion experiments were done in December 2009.

The experiments were carried out in a temperature-controlled pilot-scale diffuser. Construction and principle of operation of the diffuser and details of the diffusion experiments were reported elsewhere [46,47]. Extraction process was based on the principle of countercurrent between beet cossettes and extracting water, which is employed in the sugar beet industry [41,42]. Diffusion experiments were carried out at $T = 30\text{ °C}$ (with PEF-pretreated cossettes) and $T = 70\text{ °C}$ (with untreated cossettes). Therefore, two different juices were prepared. They will be referred below as “cold juice 30 °C” and “juice 70 °C”, correspondingly to their temperatures of diffusion.

The sugar beet cossettes were PEF treated using a pilot PEF generator (Hazemeyer, France). Electric field intensity was fixed at $E = 600\text{ V/cm}$. The monopolar pulses with pulse duration $t_i = 100\text{ }\mu\text{s}$ and pulse repetition rate $f = 200\text{ Hz}$ were used; the number of pulses n was 500. The total time of PEF treatment t_{PEF} was $n \cdot t_i = 0.05\text{ s}$. Such treatment protocol assured effective damage of the sugar beet tissue [46,49–52]. The cossettes were treated in the special section of diffuser by means of two stainless steel electrodes. The temperature elevation during PEF-treatment did not exceed 3 °C. The temperature was controlled by a Teflon-coated T-type thermocouple ($\pm 0.1\text{ K}$). More details of the PEF treatment procedure are reported elsewhere [46,47].

After diffusion, the juices were pre-filtered through a nylon mesh, frozen and stored at the temperature of -20 °C until further analysis and filtration.

2.2. Juice filtration

Filtration of the diffusion juices was carried out in the batch dead-end filtration cell Amicon 8200 (Millipore, USA) with maximal capacity of 200 ml and effective filtration surface area of $S = 2.5 \times 10^{-3}\text{ m}^2$. Two series of filtration experiments were carried

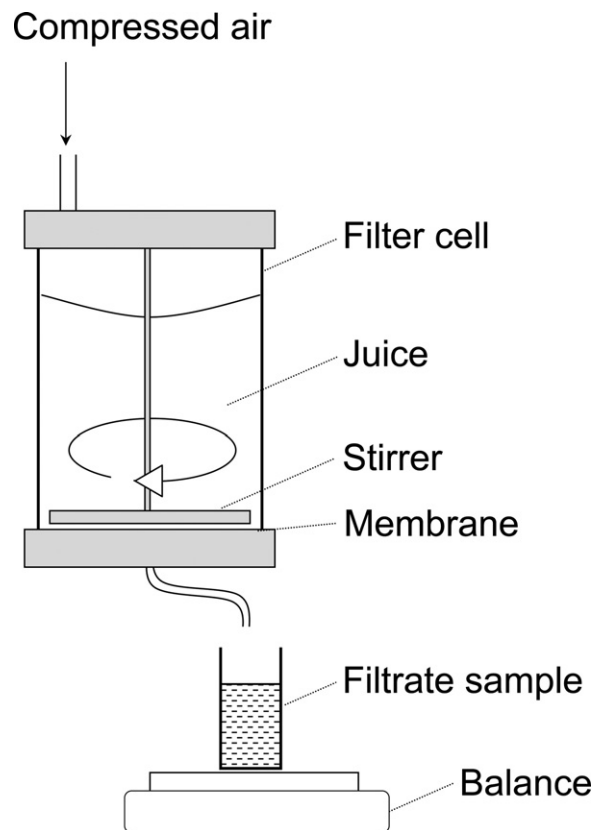


Fig. 1. Sketch of the filtration experiment.

out: without stirring and with constant stirring of the retentate. The stirring was done by means of magnetic stirrer fixed over the membrane surface and rotating at the constant rate of $\omega = 500\text{ rpm}$ (Fig. 1).

Hydrophilic polyethersulfone ultrafiltration membranes (Microdyn-Nadir GmbH, Germany) with nominal molecular weight cut-offs (MWCO) of 10, 30 and 100 kDa were used for dynamic filtration. For the unstirred filtration, the membrane with MWCO = 30 kDa was used. The membrane was new in every filtration test. Prior to filtration, membranes were washed with distilled water and resistance of membrane R_{m0} was determined from the pure water flux:

$$J = -\frac{dV}{S dt} = \frac{\Delta p}{\mu R_{m0}} \quad (1)$$

where J is the flux, V is the volume of filtrate, t is the filtration time, S is the membrane surface area, Δp is the filtration pressure and μ is the viscosity of filtrate. It was observed that clean membrane resistance was constant at various pressures within the range $\Delta p = 0.5\text{--}10\text{ bar}$. After the juice filtration, the filter-cake was removed from the membrane surface and resistance of fouled membrane R_{mf} was measured in the same manner.

For each experiment, 150 ml of the juice were used and 130 ml of filtrate were obtained during filtration. The weight of filtrate was continuously registered by means of electronic balance. The experiment was carried out in concentration mode with removing of the filtrate. The volume concentration factor VCF was calculated as

$$\text{VCF} = \frac{V_0}{V_0 - V} \quad (2)$$

where V_0 is the initial volume of juice. The final volume concentration factor was equal to 7.5.

Table 1
Properties of diffusion juices.

Diffusion juice type	Obtained at 30 °C with PEF-treatment	Obtained at 70 °C without PEF-treatment
C_{ss} (% Brix)	13.4 ± 0.3	14.8 ± 0.3
C_s (%)	12.5 ± 0.3	13.7 ± 0.1
P (%)	93.4 ± 0.2	92.8 ± 0.3
Alcohol insoluble solid (wt.%)	0.09 ± 0.03	0.22 ± 0.03
Protein content (wt.%)	0.055 ± 0.002	0.056 ± 0.002
Pectin content (wt.%)	0.003 ± 0.001	0.14 ± 0.03

Filtrate samples with volume of 5 ml were collected in small tubes and analyzed separately. Filtration experiments were carried out at the ambient temperature of 22 ± 1 °C.

2.3. Analysis of juices and filtrates

Concentration of soluble solids (Brix, %) and absorption spectra of every 5 ml filtrate sample were measured in the course of filtration.

Concentration of soluble solids was measured by means of a digital refractometer PR-32 α (Atago, Japan). The concentration of sucrose (wt.%) was determined using a polarimeter Polax 2L (Atago, Japan) after appropriate purification of the studied sample. The purity P was calculated as:

$$P = \frac{C_s}{C_{ss}} \times 100\% \quad (3)$$

where C_s is the concentration of sucrose, C_{ss} is the total concentration of soluble solids.

Light absorbance A was measured by means of spectrophotometer Biochrom Libra S32 at the wavelength $\lambda = 420$ nm. This wavelength is conventional for determination of concentration of the colored impurities and characterisation of sugar beet juice quality [23,53]. The path length of the optical cells was 10 mm. Since juices and filtrates were neutral (pH=6.8–7.0), absorbance of the samples was measured without pH adjustment.

Total concentration of colloids and polymers C_{cs} was measured in terms of alcohol insoluble solids [54]. Concentration of proteins was determined by Bradford's method [55]. Concentration of pectins was measured by enzymatic method using Pectin identification procedure (Pectin Identification, Assay Procedure, Megazyme Int., 2004). The density ρ of filtrates was measured by accurately weighting 10 ml of sample into a 10 ml picnometer. The viscosity μ was determined using a glass capillary viscosimeter. It was found that the values of density and viscosity of filtrates obtained from the “cold” juice “30 °C” are the same as those obtained from the juice “70 °C” and equal to $\rho = 1.05 \pm 0.01$ g/ml and $\mu = 1.4 \pm 0.1 \times 10^{-3}$ Pa s.

All the experiments were repeated, at least, twice, and the mean values and standard deviations were calculated. In the figures presented below, the values of error bars are equal to means standard deviations.

3. Results and discussion

3.1. Characteristics of feed juices

Chemical composition of the diffusion juices used in this study is presented in Table 1. It should be noted that the quality of the diffusion juices (concentration of soluble solids, concentration of sucrose and purity) was close to that usually obtained in sugar industry [41]. Thermal diffusion at 70 °C resulted in more intensive extraction of solutes from cossettes than in cold diffusion at 30 °C with PEF-treatment (higher values of Brix, Table 1) [47]. How-

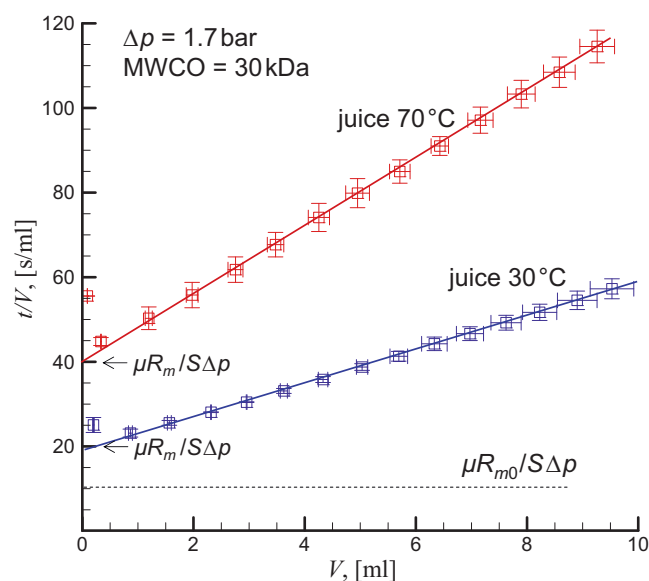


Fig. 2. Unstirred filtration curves of diffusion juices. Symbols correspond to experimental data, solid lines correspond to data fitting by Eq. (5), dashed line corresponds to resistance of clean membrane. Filtration was carried out at constant pressure $\Delta p = 1.7$ bar using membranes with MWCO = 30 kDa. Temperatures of juice extraction are shown near the symbols.

ever, hot extraction (70 °C) also resulted in thermal degradation of cellular tissue and dissolution of high molecular weight components, i.e. pectins. These compounds were more presented in the juice extracted at 70 °C (Table 1). Decrease of the diffusion temperature from 70 °C to 30 °C substantially decreases concentration of insoluble solids in the diffusion juice and almost reduces to zero concentration of pectins.

3.2. Membrane fouling during unstirred filtration

Fig. 2 shows an example of the filtration curves obtained during unstirred filtration of the diffusion juices through the membrane 30 kDa.

The Ruth-Carman's equation was applied for estimation of filter-cake resistance in unstirred filtration experiments [56].

$$\frac{t}{V} = \frac{\alpha C_{cs} \rho \mu}{2S^2 \Delta p (1 - C_{cs}/C_c)} V + R_m \frac{\mu}{S \Delta p} \quad (4)$$

where α is the specific filtration resistance of the filter-cake, C_{cs} is the weight fraction of cake-forming (colloidal and insoluble) solids in the feed juice, C_c is the weight fraction of cake-forming solids in the filter-cake, R_m is the membrane resistance, Δp is the filtration pressure, ρ and μ are the density and the viscosity of filtrate, respectively, S is the filter medium surface area, t and V are the filtration time and the filtrate volume, respectively. If concentration of the solids in the feed juice is low as compared to concentration of the solids in the filter-cake ($C_{cs} \ll C_c$), Eq. (4) may be reduced to

$$\frac{t}{V} = \frac{\alpha C_{cs} \rho \mu}{2S^2 \Delta p} V + R_m \frac{\mu}{S \Delta p} \quad (5)$$

According to Table 1, weight fraction of colloids and polymers C_{cs} in the studied juices was at the level of about 0.001 (or 0.1%). The concentration of solids in the filter-cake C_c formed during filtration of pectin and pectin containing juices varies from 5% [57] to 40% [58]. Consequently, Eq. (5) may be used for the characterisation of unstirred filtration of the studied sugar beet juices. According to Eq. (5), unstirred filtration curve must yield a straight line in coordinates t/V versus V . Values of the slope and the intercept of

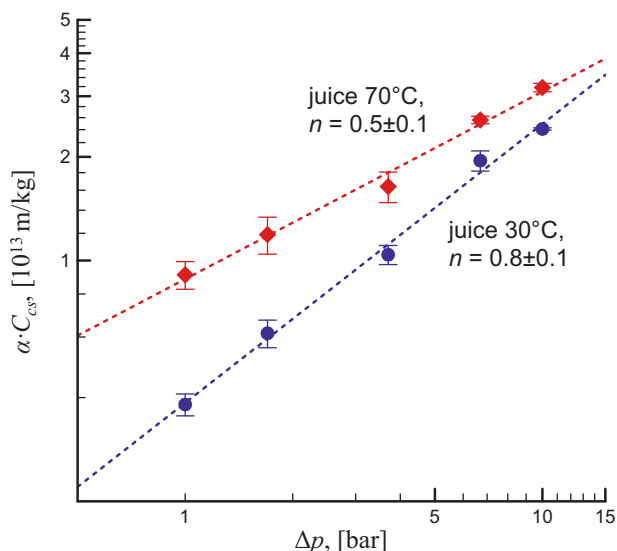


Fig. 3. Parameter $\alpha \cdot C_{cs}$ of the diffusion juices versus transmembrane pressure Δp . Symbols correspond to experimental data; lines correspond to least square fitting by means of Eq. (7).

this line are proportional to $\alpha \cdot C_{cs}$ and membrane resistance R_m , respectively.

Fig. 2 shows that filtration curves may be fitted by a straight line beyond the initial moment of filtration ($V \geq 1$ ml). It means that unstirred filtration of the extracted juices is governed by cake filtration mechanism. The same conclusion about pivotal role of the deposit or gel formation during unstirred filtration of fruit and vegetable extracts was made in [12,30,33,44,58]. It was shown that pectin and other polysaccharides are the main gel forming components of the juices.

The value of membrane resistance $R_m = 1.2 \pm 0.1 \times 10^{13} \text{ m}^{-1}$ estimated from the intercept of the filtration curves for juice “70 °C” was two times higher than that estimated for “cold” juice “30 °C” $R_m = 6.0 \pm 0.1 \times 10^{12} \text{ m}^{-1}$ (Fig. 2). Both estimated values of R_m (for juice “30 °C” and “70 °C”) were noticeably higher than the resistance of clean membrane $R_{m0} = 3.0 \pm 0.1 \times 10^{12} \text{ m}^{-1}$. This fact may reflect the presence of rapid fouling process, which steeply increases the membrane resistance during the initial few instants of filtration ($V < 1$ ml). Such rapid fouling may be attributed to adsorption of colloidal species, internal pore blocking or pore plugging processes that were observed during filtration of various juices [2,5,7,30,59]. It was reported that polysaccharides, particularly pectins, which are present in the extracted juices, may attach to the membrane surface or pore walls and cause irreversible membrane fouling [7,30,60]. In the present study irreversible fouling was estimated from measurements of the membrane resistance after removal of the filter-cake R_{mf} . It was found that irreversible fouling during filtration of “cold” juice “30 °C” increased the membrane resistance by 50% ($R_{mf}/R_{m0} = 150 \pm 10\%$). Filtration of juice “70 °C”, which has much higher concentration of fouling colloids and polymers, increased the membrane resistance by 100% ($R_{mf}/R_{m0} = 200\% \pm 20\%$).

The resistance of formed deposit R_g is determined by the value of $\alpha \cdot C_{cs}$ [61]:

$$\alpha \cdot C_{cs} = \frac{R_g S}{V \rho} \quad (6)$$

Therefore, the value of $\alpha \cdot C_{cs}$ is a measure of membrane fouling due to deposit formation (the higher is the value of $\alpha \cdot C_{cs}$ the higher is the fouling). Fig. 3 presents the values of $\alpha \cdot C_{cs}$ estimated for juices “30 °C” and “70 °C” from the unstirred filtration curves using Eq. (5).

It is seen that the fouling during unstirred filtration of juice “70 °C” is noticeably higher than during filtration of juice “30 °C”.

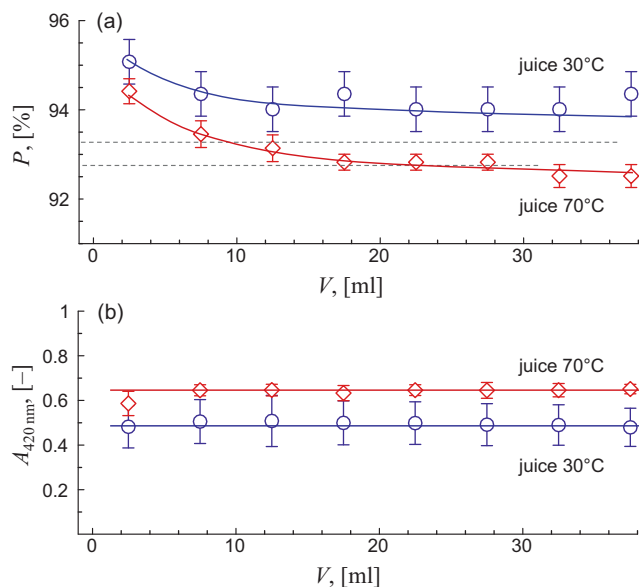


Fig. 4. Dependence of filtrate purity P (a) and absorbance A (b) on filtrate volume V during unstirred filtration of sugar beet juices extracted at “70 °C” and “30 °C”. Dashed lines correspond to the purity of feed juices. Filtration was carried out at the constant pressure $\Delta p = 1.7$ bar using membranes with MWCO 30 kDa.

Fouling also increases with transmembrane pressure (Fig. 3). Since C_{cs} is constant for a given juice, dependence of $\alpha \cdot C_{cs}$ on applied pressure may be described by empirical equation

$$\alpha \cdot C_{cs} = \alpha_0 (\Delta p)^n \cdot C_{cs} \quad (7)$$

where α_0 and n are constants [39]. Fitting of the experimental data by Eq. (7) yields $\alpha_0 \cdot C_{cs} = (3.2 \pm 0.3) \times 10^{12} \text{ m/kg}$ and $n = 0.8 \pm 0.1$ for juice “30 °C” and $\alpha_0 \cdot C_{cs} = (7.2 \pm 0.7) \times 10^{12} \text{ m/kg}$ and $n = 0.5 \pm 0.1$ for juice “70 °C”. The calculated values of n suggest that deposit layer formed during filtration of diffusion sugar beet juices is compressible. The calculated values of n are in agreement with the values reported for protein, pectin and various natural juices [39,44,58,62,63]. The ratio between values of $\alpha_0 \cdot C_{cs}$ calculated for juices “70 °C” and “30 °C” (≈ 2.3) is close to the ratio between experimentally measured concentrations of colloidal substances in the studied juices (≈ 2.4). It suggests that better filterability of cold juice “30 °C” can be explained by lower concentration of pectin and other colloidal substances. The estimated values of specific filtration resistance α for juices “30 °C” and “70 °C” were approximately equal: $\alpha \approx (5.4 \pm 0.6) \times 10^{15} \text{ m/kg}$ (at the pressure of 2 bar). The estimated value of α was close to that reported for various pectin containing juices [28,33,39,44,58].

3.3. Purity and absorbance of filtrate obtained by unstirred filtration

Fig. 4 presents the evolutions of filtrate purity P and absorbance A during unstirred filtration of diffusion juices. The filtrate purity is higher at the beginning of filtration (Fig. 4a). However it rapidly decreases and approaches the purity of feed juice. Initial increase of filtrate purity may be explained by formation of deposit layer of colloidal compounds on the membrane surface. Mobility of low molecular weight compounds in the deposit layer and membrane pore volume is reduced as compared to their mobility in bulk solution [33,58,64]. It results in slower transmission of the low molecular weight impurities through the deposit and membrane to filtrate and higher purity of the first portions of filtrate [33,58,64]. However, after certain time of filtration, concentration of rejected impurities on the membrane surface increases and deposit layer

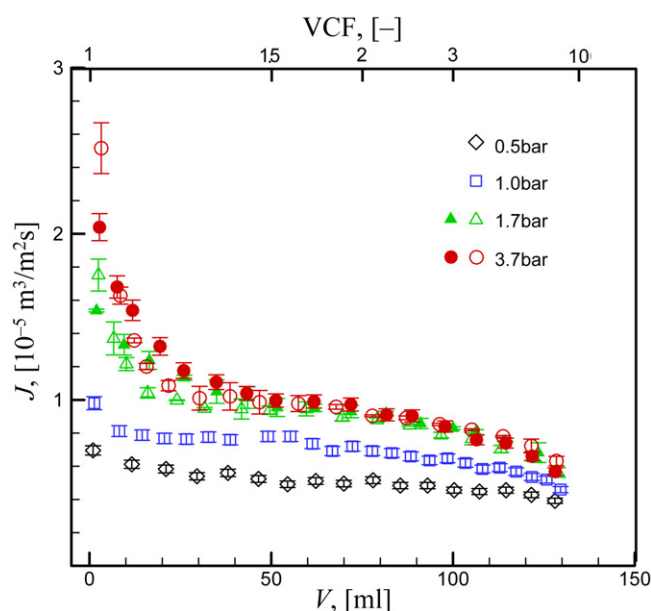


Fig. 5. Dependence of filtrate flux J versus filtrate volume V and volume concentration factor VCF during filtration with stirring of sugar beet juices extracted at 30 °C (open symbols) and 70 °C (filled symbols). The membrane MWCO = 30 kDa, stirring velocity $\omega = 500$ rpm, filtration pressure Δp is shown near the symbols.

becomes saturated with solutes. Therefore, transmission of impurities into filtrate increases and purity of filtrate decreases. The same evolution of filtrate purity was reported for filtration of various natural and synthetic juices [29,33,58].

In contrast to purity P , absorbance of filtrate A remains constant during unstirred filtration of extracted juices (Fig. 4b). Hence, the role of deposit formation in separation of juice colorants is relatively minor. Note that filtrate obtained from the “cold” juice “30 °C” has higher purity and lower absorbance as compared to filtrate obtained from the juice “70 °C” (Fig. 4). It may be explained by better quality of the juice extracted at lower temperature with application of PEF-treatment (lower concentration of impurities and colorants) [47,48].

It is known that coloration of sugar beet juices may be caused by different compounds: melanins, melanoidins, caramels and HADP [23,41,53]. These colorants are formed due to various chemical reactions between extracted components of the cellular juice and their formation is accelerated by the temperature increase [23,53]. Hence, decrease of extraction temperature from 70 to 30 °C prevents or retards formation of colorants and results in better quality of juices and filtrates. More precise determination of colorants nature requires additional analysis.

3.4. Membrane fouling during filtration with stirring

Filtration curves obtained during filtration of the diffusion juices under constant velocity stirring are shown in Fig. 5. All the filtration curves presented in Fig. 5 follow typical trend for dynamic filtration of diluted colloidal suspensions. The flux steeply decreases on the beginning of filtration (corresponding to $V \approx 0$ –35 ml) and then slowly decreases with increase of V and VCF . It is generally assumed that fast initial flux decrease corresponds to membrane fouling (internal fouling, pore blocking or deposit formation). Subsequent stage corresponds to stationary filtration through the fouled membrane and filtrate flux is determined by resistance of fouled membrane. Fig. 5 shows that the filtrate flux increases with the applied pressure on the initial stage of filtration. However, beyond the initial stage, the values of flux are less dependent on the pressure. It suggests that intensity of membrane fouling increases

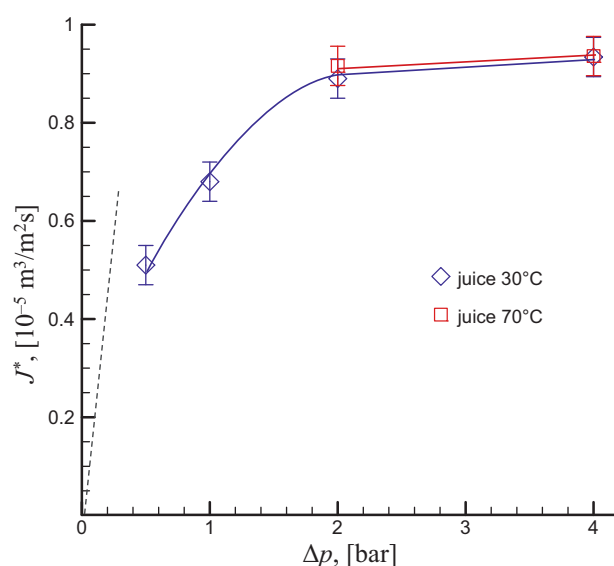


Fig. 6. Variation of quasi-stationary flux J^* (measured at $VCF=2$) with transmembrane pressure (MWCO = 30 kDa, $\omega = 500$ rpm). Dashed line corresponds to filtration through the clean membrane.

with applied pressure. On the initial stage of filtration, higher pressure results in more rapid decrease of flux that may be explained by faster transport of colloidal and polymer species to the membrane surface and faster formation of deposit. The flux remains almost constant after the deposit formation except for the final stage of filtration corresponding to high VCF . Total resistance of fouled membrane increases with pressure and thus formation of thicker deposit counteracts to the increase of filtration rate.

Fig. 6 shows pressure dependence of filtration rate J^* measured in a quasi-stationary stage of filtration ($V = 75$ ml, $VCF = 2$). It is seen that for juice “30 °C”, the flux J^* becomes higher with pressure increase in the range $\Delta p = 0$ –1.7 bar, then the flux J^* reaches a plateau and becomes nearly constant at $\Delta p > 1.7$ bar. Similar curves with plateau were observed for various natural and synthetic juices [1,2,6,13,60]. According to the gel polarisation model, this plateau corresponds to the situation when deposit layer is formed over the entire membrane surface and filtration is controlled by mass-transfer controlled regime [57]. In this regime, the transport of colloidal and polymer foulants to the surface of deposit is balanced by shear-induced back-transport of the foulants to the bulk solution [57]. Depending on interaction between colloids and membrane surface, the deposit may be both rigid and attached to the membrane, or it can be flowable. At lower pressure, the membrane surface may be partly uncovered by deposit and filtration may be controlled by pressure regime (which is characterised by proportionality between the flux and pressure) [57]. The deposit-free part of membrane can be situated in the region with highest shear stress (that is an outer region of membrane in the stirred cell) [65].

When filtration is operated in the mass-transfer controlled regime, evolution of the stationary flux may be described using the equation

$$J = k \ln \left(\frac{C_c}{C_{cs}} \right) \quad (8)$$

where C_c is the concentration of foulant in the deposited layer, C_{cs} is the concentration of foulant in the retentate, k is the mass transfer coefficient, which depends on the foulant diffusivity [8,28,57]. A mass balance equation for foulant is

$$C_{cs} \cdot (V_0 - V) = C_{cs0} \cdot V_0 \quad (9)$$

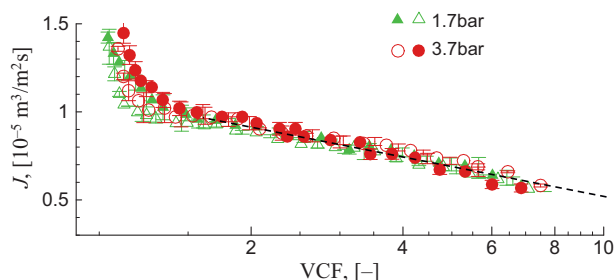


Fig. 7. Filtrate flux versus VCF. Open symbols correspond to cold “juice 30 °C”, filled symbols correspond to “juice 70 °C”, corresponds to fitting of experimental data by Eq. (10). MWCO = 30 kDa, ω = 500 rpm.

where C_{cs0} is the initial concentration of foulant. Combination of Eqs. (2), (8) and (9) gives

$$J = k^* - k \ln(\text{VCF}) \quad (10)$$

where k^* is the constant, $k^* = k \ln(C_c/C_{cs0})$. Eq. (10) suggests that in the mass-transfer controlled filtration, dependence of the stationary flux versus VCF must yield a straight line with the pressure-invariant parameters k^* and k .

Fig. 7 presents the dependences of flux versus logarithm of VCF obtained for filtration of sugar beet juices at the pressures $\Delta p = 1.7$ and 3.7 bar. The figure shows that beyond the initial stage of filtration (i.e., at $\text{VCF} < 1.5$) filtration curves may be fitted by linear Eq. (10) with correlation coefficient $r^2 > 0.95$. The fitting parameters were pressure-invariant in accordance with mass-transfer mechanism of filtration.

It should be noted that the rates of filtration of the sugar beet juices extracted at 70 °C and at 30 °C were approximately equal during the stationary filtration stage in spite of much lower concentration of colloidal impurities (especially, pectins) in the juice extracted at 30 °C. It may be speculated that residual impurities contained by the juice extracted at 30 °C (e.g., proteins) also play an important role in formation of the deposited layer. It was shown in the literature [34] that depectinization of fruit juice can not prevent the membrane fouling and polarization, since the polarized layer is formed from the residual pectin molecules and other colloidal substances. Also, it was shown that maximal steady-state flux may be attained only after removal of both pectins and proteins from the filtered juice [34].

Fig. 8 presents normalized filtrate fluxes on the initial stage of filtration of sugar beet juices “30” and “70 °C”. It shows that normalized fluxes steeply decrease from 1 to 0.3–0.4 after filtration of the first portions of filtrate. Similar immediate fouling of membrane during filtration was reported in [5,30]. It suggests fast pore blocking and formation of polarized layer [5,30]. Initial flux decline

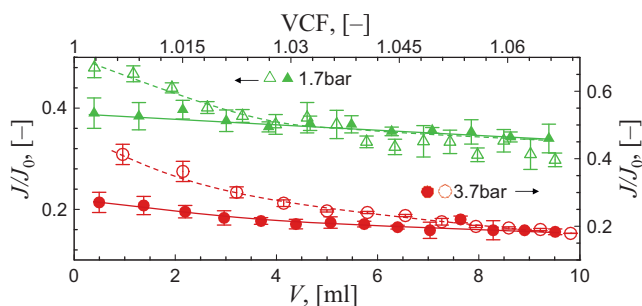


Fig. 8. Normalized filtrate flux versus filtrate volume and VCF at $\Delta p = 1.7$ bar (triangles) and $\Delta p = 3.7$ bar (circles). Open symbols correspond to the juice obtained at 30 °C, filled symbols correspond to the juice obtained at 70 °C (MWCO = 30 kDa, ω = 500 rpm).

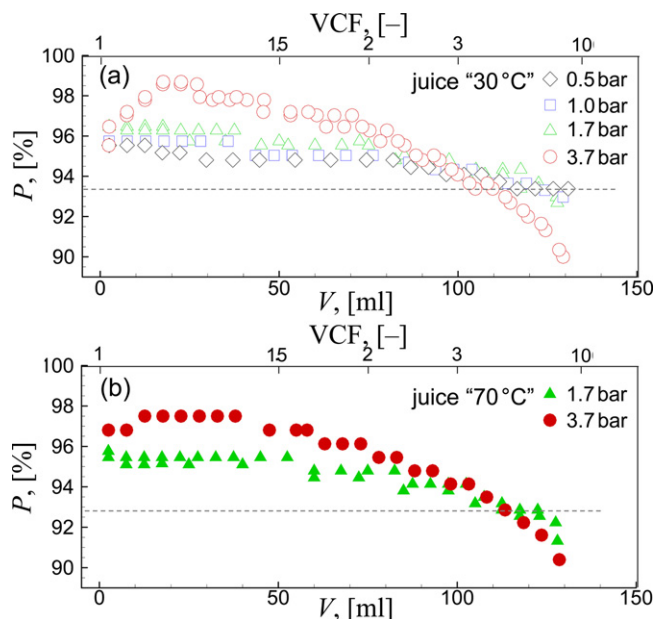


Fig. 9. Purity of filtrate versus filtrate volume and VCF during filtration with stirring of the sugar beet juice extracted at 30 °C (a) and 70 °C (b) filtered at various pressures. Dashed lines correspond to the purity of feed juices. Membrane MWCO = 30 kDa. The values of error bars are of the order of symbol size.

is more drastic during filtration of juice “70 °C” as compared to juice “30 °C”. This may be explained by higher concentration of foulant (colloids and polymers) in the juice extracted at higher temperature.

3.5. Purity of filtrate obtained during filtration with stirring

Fig. 9 presents the data on evolution of filtrate purity during filtration with stirring of sugar beet juices. Purity was calculated using Eq. (3) from concentration of soluble solids and sucrose in the filtrate. It should be noted that rejection of sucrose was not observed. It is seen that the purity of filtrate is substantially higher than the purity of initial juices (presented by dashed lines). Therefore, stirred filtration of sugar beet juices through the membrane with MWCO = 30 kDa results in efficient purification of juice and removal of non-colloidal (low molecular weight) impurities. It should be noted that the observed values of purity were higher than purity of ultrafiltrates of sugar beet juices reported in [3,24–26], probably, due to better quality of the diffusion juices used in this study (Table 1).

Comparison of the average filtrate purity (at $\text{VCF} = 2$, $\Delta p = 1.7$ bar) has shown that the average purity of the filtrate obtained from juice extracted at 30 °C was $95.3 \pm 0.4\%$, while the average purity of the filtrate obtained from juice extracted at 70 °C was $94.7 \pm 0.4\%$. This difference may be explained by higher purity of initial juice obtained by extraction at 30 °C. Purity of the filtrate obtained in stirred filtration was noticeably higher than purity of the filtrate obtained in unstirred filtration (Figs. 4a and 9). It may be explained by higher rejection of impurities during the stirred filtration due to their sweeping from the membrane surface by shear flow [66,67]. The fraction of impurities swept away from the membrane or deposit surface increases with increase of the stirring speed. This implies that permeation of impurities into the filtrate may be reduced by stirring of the filtered juice [66,67].

Fig. 9 shows that purity of filtrate increases with transmembrane pressure. The increase of filtrate purity with applied pressure was observed previously during filtration of various juices and extracts [1,2,22,29]. The increase of purity at higher pressure is probably

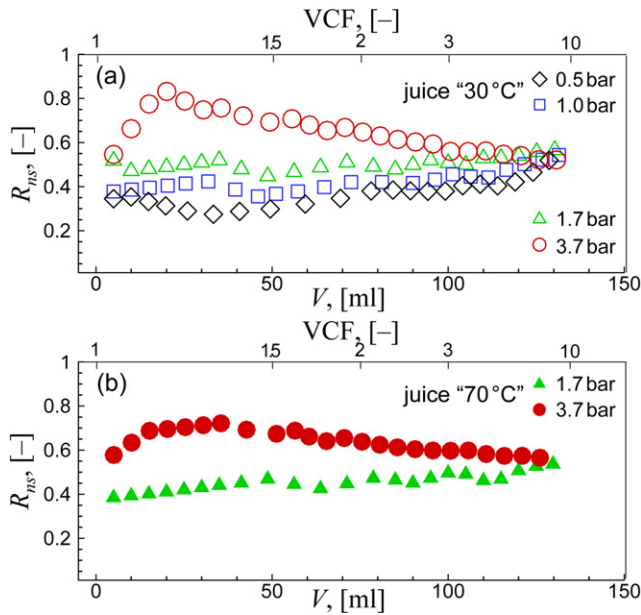


Fig. 10. Coefficient of rejection of non-sucrose compounds R_{ns} versus V and VCF: (a) filtration of juice “30 °C” and (b) filtration of juice “70 °C”. Membrane MWCO = 30 kDa. The values of error bars are of the order of symbol size.

associated with development and compression of the polarized layer of colloidal particles and polymer molecules, which may serve as a dynamic membrane and enhance separation of impurities [18,29,31]. The effect of polarized layer on purity is most pronounced at higher pressure $\Delta p = 3.7$ bar, when polarized layer is formed over the entire membrane surface.

Fig. 9 shows that beyond the initial stage of filtration the filtrate purity gradually decreases with increase of filtrate volume. This may be explained by accumulation of impurities in the retentate and by decrease of retentate purity. Consequently, the purity of filtrate may decrease even at constant coefficient of rejection of non-sucrose compounds R_{ns} , determined as

$$R_{ns} = 1 - \frac{C_f}{C_r} \quad (11)$$

where C_f and C_r are concentrations of non-sucrose impurities in the filtrate and retentate, respectively. The value of R_{ns} may vary from $R_{ns} = 0$ (that means the absence of purification during filtration) to $R_{ns} = 1$ (means complete rejection of impurities and maximal purification of filtrate).

In the experiments with closed batch filtration cell, the value of R_{ns} may be determined without the measurement of C_r during filtration. It may be calculated from the dependence of concentration of impurities in the filtrate C_f versus VCF using the mass balance equation for impurities:

$$C_0 V_0 = C_r (V_0 - V) + \int_0^V C_f dV \quad (12)$$

where C_0 is the concentration of impurities in the feed juice, V_0 is the volume of juice used for filtration. Combination of Eqs. (11) and (12) yields

$$R_{ns} = \frac{1 - C_f (V_0 - V)}{C_0 V_0 - \int_0^V C_f dV} \quad (13)$$

Fig. 10 presents dependence of R_{ns} calculated using Eq. (13) versus filtrate volume and VCF. It was observed that rejection coefficient was almost constant during the filtration (except of filtration carried out at $\Delta p = 3.7$ bar). The same conclusion about constancy of R_{ns} during filtration of juices and extracts was done in

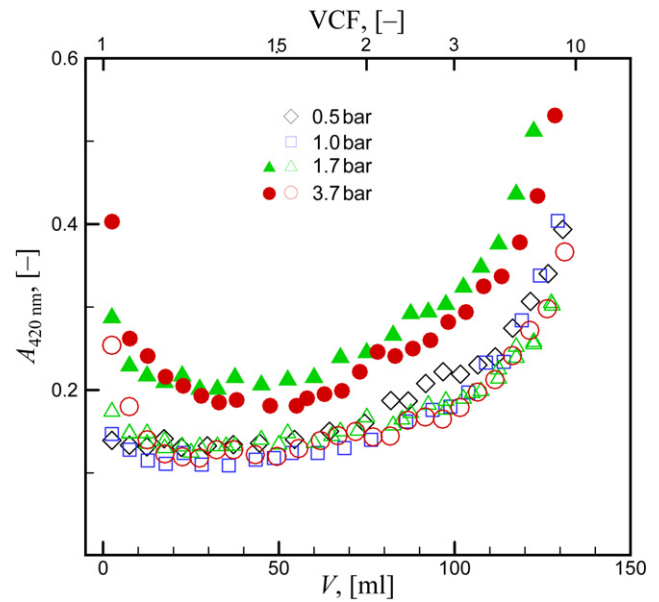


Fig. 11. Absorbance of filtrate A (at $\lambda = 420$ nm) versus filtrate volume and VCF during the stirred filtration of sugar beet juices extracted at 30 °C (open symbols) and 70 °C (filled symbols). Transmembrane pressure is shown near the symbols. Membrane MWCO = 30 kDa. The values of error bars are of the order of symbol size.

[18,23,68]. The value of R_{ns} increases with transmembrane pressure in correspondence with the previously reported studies [1,2,22,29]. Maximal value of R_{ns} was observed for filtration at high pressure $\Delta p = 3.7$ bar, when polarized layer was formed over the entire membrane surface and resistance of polarized layer was maximal (Fig. 10). The polarised layer acted as a dynamic membrane and separation performance of this dynamic membrane was of the same order of magnitude as separation performance of ultrafiltration membrane with MWCO = 30 kDa. Therefore, steep increase of rejection during the initial stage of filtration at $\Delta p = 3.7$ bar corresponds to formation of dynamic membrane (polarised layer). Next gradual decrease of R_{ns} may be explained by higher concentration of impurities in the polarised layer as compared to bulk concentration of impurities. Higher concentration of impurities in the polarised layer results in lowering of observed rejection (rejection calculated by Eq. (13)), even if real rejection coefficient remains constant [69].

3.6. Absorbance of filtrate obtained during filtration with stirring

Fig. 11 presents evolution of filtrate absorbance during stirred ultrafiltration of diffusion juices. Absorbance of filtrate obtained from juice “30 °C” was noticeably lower than absorbance of filtrate obtained from juice “70 °C” (e.g., the average absorbance of filtrate obtained from juice extracted at 30 °C was 0.143 ± 0.005 , while the average absorbance of filtrate obtained from juice extracted at 70 °C was 0.228 ± 0.011 at VCF=2, $\Delta p = 1.7$ bar). This may be explained by lower concentration of colorants in the feed juice extracted at “30 °C” (absorbance of unfiltered juices “30 °C” was 1.05 ± 0.04 , while absorbance of unfiltered juice “70 °C” was 1.47 ± 0.05). Absorbance of filtrate decreased during the initial stage of filtration ($V \leq 30$ ml). Initial decrease of absorbance was observed for every experiment within the studied pressure range. At the following stage of filtration, absorbance of filtrate increased with increase of VCF. Such increase of absorbance of the filtrate may be explained by continuous increase of colorant concentration in the retentate. Unlike the purity, the value of absorbance was practically independent of the applied pressure (except of initial stage of filtration). It suggests that formation of polarised layer or deposit layer on the membrane surface had only minor effect on separation

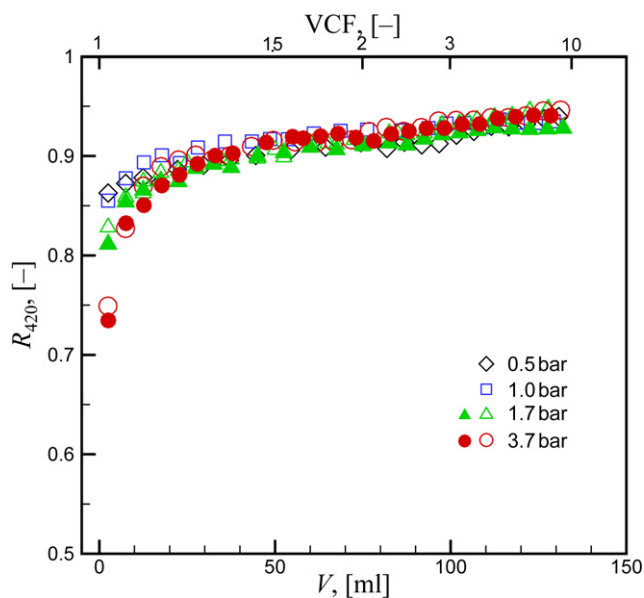


Fig. 12. Coefficient of rejection of colorants R_{420} versus V and VCF during filtration of sugar beet juices extracted at 30 °C (open symbols) and 70 °C (filled symbols). Membrane MWCO = 30 kDa. The values of error bars are of order of symbol size.

performance against colorants during ultrafiltration of sugar beet juices.

Since absorbance is directly proportional to concentration of colorants, the coefficient of rejection of colorants R_{420} may be estimated from absorbance A versus V using equation analogous to Eq. (13):

$$R_{420} = \frac{1 - A(V_0 - V)}{A_0 V_0 - \int_0^V A dV} \quad (14)$$

where A_0 is the absorbance of non-filtered juice.

Fig. 12 presents dependence of R_{420} calculated by Eq. (14) versus V and VCF. The coefficient of rejection of colorants absorbing at the wavelength of 420 nm is pressure invariant. The value of the coefficient of rejection is equal to $R_{420} \approx 0.9$ –0.95; therefore, filtration results in efficient removal of colored compounds from the sugar beet juice. Rejection decreases during the initial stage of filtration, probably, due to internal fouling of the membrane. It was shown previously that internal fouling of ultrafiltration membranes decreases their MWCO and enhances their separation performance against low molecular weight compounds [30,31].

3.7. Filtration with various membranes

Fig. 13 presents evolution of the flux during ultrafiltration of the sugar beet juices through membranes with various MWCO. Though permeability of pristine membranes strongly depends on MWCO, the filtration rate of the sugar beet juices is less sensitive to membrane properties (Fig. 13). Filtrate flux steeply decreases at the beginning of filtration ($V \leq 30$ ml) and then slowly declines during subsequent filtration stage. Fast flux decline may be explained by internal membrane fouling [30] and formation of polarised layer [13,32]. When the membrane fouling is completed, filtration rate is limited by resistance of polarized layer. The effect of MWCO or membrane permeability on the steady state flux is minor. Same observation was reported previously for filtration of depectinized fruit juice through the membranes with MWCO within the range of 10–100 kDa [32].

Fig. 14 shows purity of filtrates (calculated using Eq. (3)) obtained during filtration of sugar beet juices through the membranes with various MWCO. Purity of filtrate decreases as the

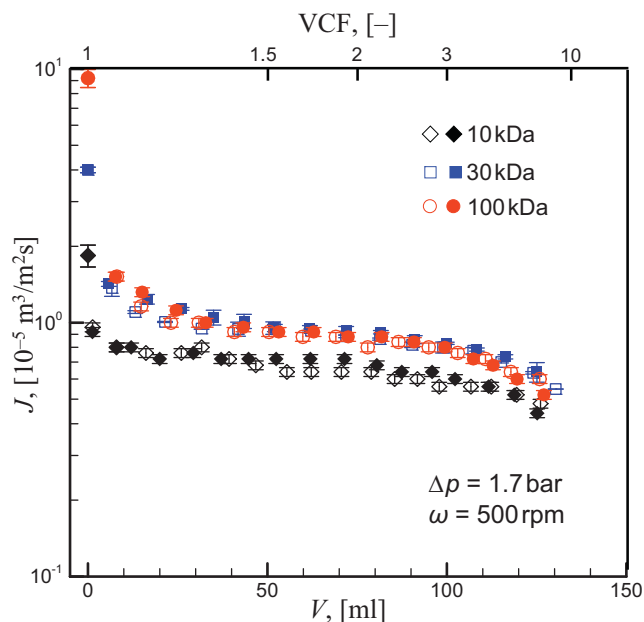


Fig. 13. Filtrate flux J versus filtrate volume V and volume concentration factor VCF for sugar beet juices extracted at 30 °C (open symbols) and 70 °C (filled symbols). The values of membrane MWCO are shown near the symbols. Filtration was carried out under constant stirring $\omega = 500$ rpm and pressure $\Delta p = 1.7$ bar.

membrane pore size increases (Fig. 14). This may be explained by better retention of low molecular weight impurities. Purity also gradually decreases with VCF during the filtration. Decrease of purity during filtration is related to increase of the concentration of impurities in the retentate with increase of VCF.

Fig. 15 presents absorbance of filtrate obtained using various membranes. Absorbance of filtrate decreases with increase of membrane's MWCO due to rejection of smaller solutes by the membrane with smaller pore size. The average absorbance of filtrates obtained from the juice "30 °C" was equal to 0.046 ± 0.002 ,

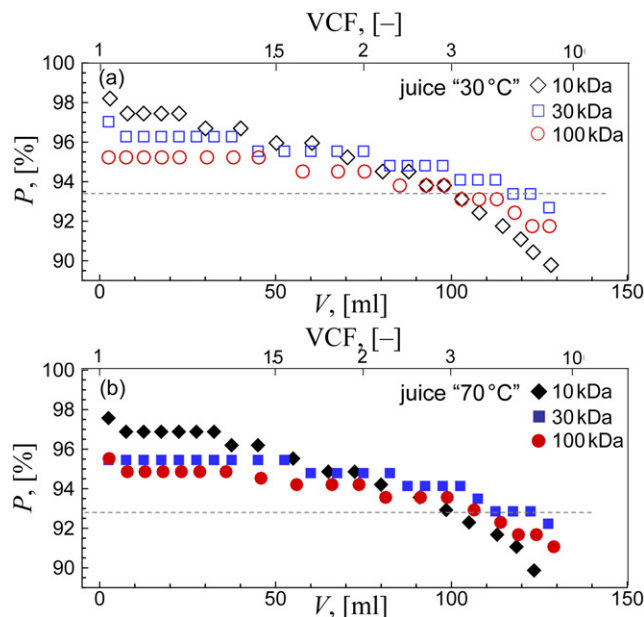


Fig. 14. Purity of filtrate samples obtained during filtration of sugar beet juices extracted at 30 °C (a) and 70 °C (b). Dashed lines correspond to the purity of feed juices. The values of membrane MWCO are shown near the symbols. Filtration was carried out under constant stirring $\omega = 500$ rpm and pressure $\Delta p = 1.7$ bar. The values of error bars are of the order of symbol size.

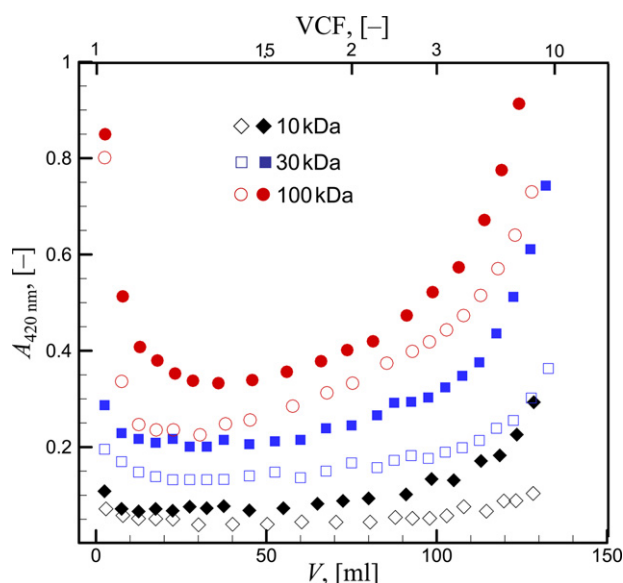


Fig. 15. Absorbance of filtrate samples obtained during filtration of sugar beet juices extracted at 30 °C (open symbols) and 70 °C (filled symbols). The values of membrane MWCO are shown near the symbols. Filtration was carried out under constant stirring $\omega = 500$ rpm and pressure $\Delta p = 1.7$ bar. The values of error bars are of the order of symbol size.

0.143 ± 0.005, and 0.292 ± 0.011 for membranes with MWCO equals 10 kDa, 30 kDa, and 100 kDa, respectively (at VCF = 2). For the juice “70 °C”, the average absorbance of filtrates (at VCF = 2) was equal to 0.076 ± 0.004, 0.228 ± 0.011, and 0.392 ± 0.019 for membranes with MWCO equals 10 kDa, 30 kDa, and 100 kDa, respectively. Absorbance of juice extracted at 30 °C is lower than absorbance of juice extracted at 70 °C, probably, due to higher concentration of colorant in the juice extracted at higher temperature [47]. Lower concentration of colorants in filtrate obtained from the juice extracted at 30 °C may also suggest higher average molecular weight of colorants in this juice that facilitates their separation.

Fig. 16 shows profiles of the coefficient of rejection of colorants R_{420} estimated using Eq. (14) for filtration of sugar beet juices through the membranes with various MWCO. The coefficient of

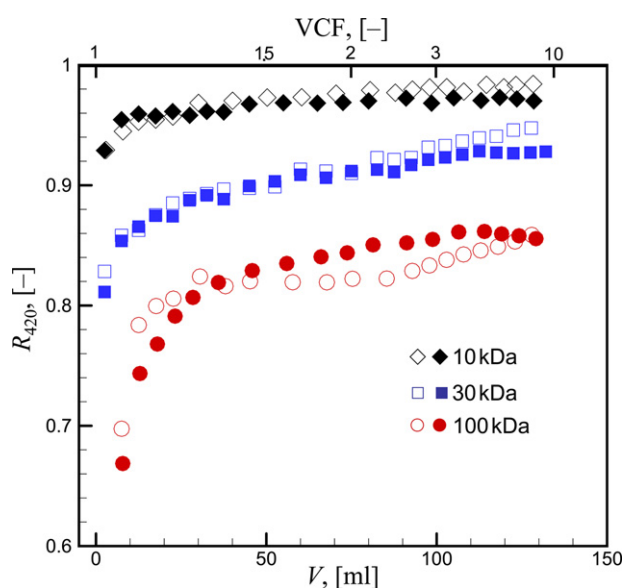


Fig. 16. Coefficient of rejection of colorants R_{420} versus V and VCF during filtration of juice “30 °C” (open symbols) and juice “70 °C” (filled symbols). Membranes MWCO are shown near the symbols. The values of error bars are of the order of symbol size.

rejection increases during filtration of the first portions of filtrate and remains almost constant during the subsequent stage of filtration (at VCF = 1.5–7.5). This behaviour may be caused by internal membrane fouling (pore narrowing) at the beginning of filtration that decreases the membrane MWCO [30,31].

The data presented in Figs. 14–16 suggest that using of membranes with lower MWCO is more efficient for purification and decolorization of the sugar beet juice since it results in lower concentration of low molecular weight impurities and colorants in the filtrate. Filtrate purity and coloration are not directly related to the pore size of pristine membrane. However, purity and coloration depend on the pore size of the fouled membrane. The data presented in Figs. 11 and 12 suggest that the presence of polarised layer on the membrane surface has rather minor effect on the retention of colorants. However, the polarised layer has strong influence on the filtrate flux.

4. Conclusions

PEF-assisted aqueous extraction of sugar beet at $T = 30$ °C results in a noticeably lower concentration of colloidal and polymer foulants in the diffusion juice as compared to the diffusion of juice obtained by conventional method at $T = 70$ °C. Reduced concentration of foulants (especially of pectins) in the juice obtained at 30 °C results in higher filterability measured upon unstirred dead-end ultrafiltration. The better filterability is explained by reduced formation of deposit.

However, under constant stirring conditions, filtration rate of juice extracted at 30 °C was nearly equal to filtration rate of juice extracted at 70 °C. Filtration was limited by formation of polarized gel layer from residual colloidal foulants (at high transmembrane pressure) and by internal membrane fouling (at low transmembrane pressure).

Filtration with stirring of sugar beet juice obtained by PEF-assisted low temperature diffusion resulted in better filtrate quality: higher purity and lower coloration. It was observed that formation of polarized layer had only minor effect on the filtrate purity. However, the filtrate purity and coloration were greatly affected by MWCO of the membrane used: filtrate purity increased and coloration decreased with decrease of MWCO from 100 to 10 kDa. High rejection of impurities and colorants was attained after the initial membrane fouling.

Acknowledgements

Authors thank the society Tereos for the providing of sugar beets during the campaign 2009. Authors also thank Dr. N.S. Pivovarova for her help with preparation of the manuscript and Dr. O. Bals for useful discussions.

Nomenclature

Notation

A	light absorbance, dimensionless
C_c	concentration of colloids and polymers in filter cake, %
C_{cs}	concentration of colloids and polymers in juice, %
C_f	concentration of impurities in filtrate, %
C_r	concentration of impurities in retentate, %
C_s	concentration of sucrose, %
C_{ss}	total concentration of soluble solids, %

J	filtrate flux, $\text{m}^3/(\text{m}^2 \text{ s})$
P	purity, %
Δp	filtration pressure, Pa
R_g	filtration resistance of deposited layer, m^{-1}
R_m	resistance of filtration membrane, m^{-1}
R_{m0}	resistance of pristine membrane, m^{-1}
R_{mf}	resistance of fouled membrane, m^{-1}
R_{ns}	coefficient of rejection of non-sucrose, dimensionless
R_{420}	coefficient of rejection of colorants, dimensionless
S	membrane surface area, m^2
t	filtration time, s
T	diffusion temperature, $^\circ\text{C}$
V	volume of filtrate, m^3

Greek letters

α	specific cake resistance, m/kg
λ	wavelength, nm
μ	viscosity of filtrate, Pa s
ρ	density of filtrate, kg/m^3
ω	stirring rate, rpm

Abbreviations

MWCO	molecular weight cut-off
PEF	pulsed electric field
VCF	dimensionless volume concentration factor

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